REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*
31-10-2005

2. REPORT TYPE
Final Report

3. DATES COVERED *(From – To)*
14 September 2001 - 29-Mar-06

4. TITLE AND SUBTITLE

Dielectric Non-Destructive Testing of Adhesively Bonded and Composite Aircraft Structures

5a. CONTRACT NUMBER
F61775-01-C0005

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

Professor Richard Pethrick

5d. PROJECT NUMBER

5d. TASK NUMBER

5e. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
University of Strathclyde
295 Cathedral Street
Glasgow G1 1XL
United Kingdom

8. PERFORMING ORGANIZATION REPORT NUMBER

N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

EOARD
PSC 821 BOX 14
FPO 09421-0014

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
SPC 01-4055

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

This report results from a contract tasking University of Strathclyde as follows: The contractor will investigate dielectric non-destructive inspection of adhesive bonds and bonded structures in the following areas:
- to explore the potential of dielectric measurements for the study of boron fiber/aluminum adhesive bonded structures used for in-situ repairs of aircraft.
- to evaluate the potential of the dielectric technique to the study of carbon fiber composite/aluminum adhesive bonded structures currently used to repair damage in aircraft.
- to explore the potential of the method by examination of real airframe structures.
- to characterize film adhesives and the state of cure in adhesive materials.

15. SUBJECT TERMS
EOARD, Aging Aircraft, Non-destructive inspection, Polymer Chemistry

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18, NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			MATTHEW MORGAN, Lt Col, USAF
UNCLAS	UNCLAS	UNCLAS	UL	12	**19b. TELEPHONE NUMBER** *(Include area code)* +44 (0)20 7514 4505

Dielectric Non-Destructive Testing of Adhesively Bonded and Composite Aircraft Structures

A final report for phase 3 of contract F61775-01-C0005

for

European Office of Aerospace Research and Development

by

RA Pethrick, D Hayward & B McConnell
Department of Pure & Applied Chemistry
University of Strathclyde
Thomas Graham Building
295 Cathedral Street
Glasgow, Scotland
UK

Introduction

This is nominally the final report for the third phase of the project as outlined in the initial proposal but because the PhD student started one year later into the project than originally planned some of the experiments are still ongoing and this report details the work in progress. Some of the theoretical work has also been rescheduled so that the conclusions will be presented in the overall final report.

1. Test joints

3 sets of bonded joints have been aged in water baths to the point that they appeared to display a plateau in their behaviour. They are now undergoing desorption to see how much of their original properties are recovered

Aluminium/Vantico Epibond 1590

Aluminium plates bonded with a new Vantico rubber toughened adhesive Epibond 1590 have been immersed in water at 50^{o}C and 75^{o}C.
The permittivity at 10kHz and 3MHz is plotted in Figure 1and Figure 2. For this particular system the data isn’t truly reaching an equilibrium value, rather the scatter in the data at long times has become very large. Clearer evidence of an equilibrium state is shown in the time domain data Figure 3 where the time value of the first major peak is reaching a stable value. The poor quality of the trace at long times is consistent with the scatter in the frequency domain data, both are symptomatic of increasing inhomogeneity in the joints.
The mechanical test data of the joints over the same period, Figure 4, shows that the initial joints were probably not fully cured in that the exposure didn’t show the usual monotonic decrease in properties with time, rather there is evidence of stress redistribution, and possibly further cure during the exposure. This again is consistent with the dielectric data in that the initial poor uniformity of the adhesive cure shows up at long times in non-uniform dielectric behaviour.
The joints are now undergoing desorption during which time the dielectric behaviour will be monitored and the final mechanical tests carried out.

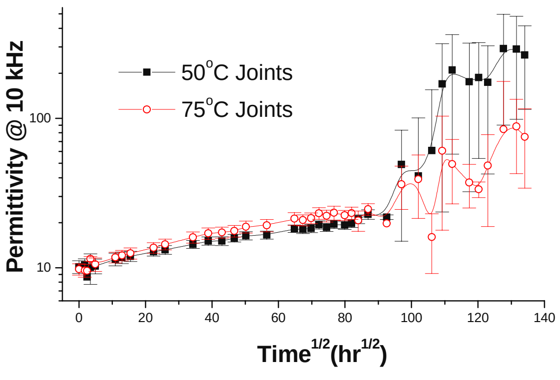

Figure 1 10kHz permittivity for Epibond

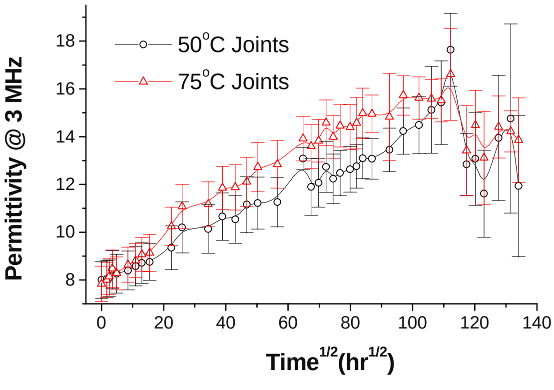

Figure 2 3MHz permittivity for Epibond

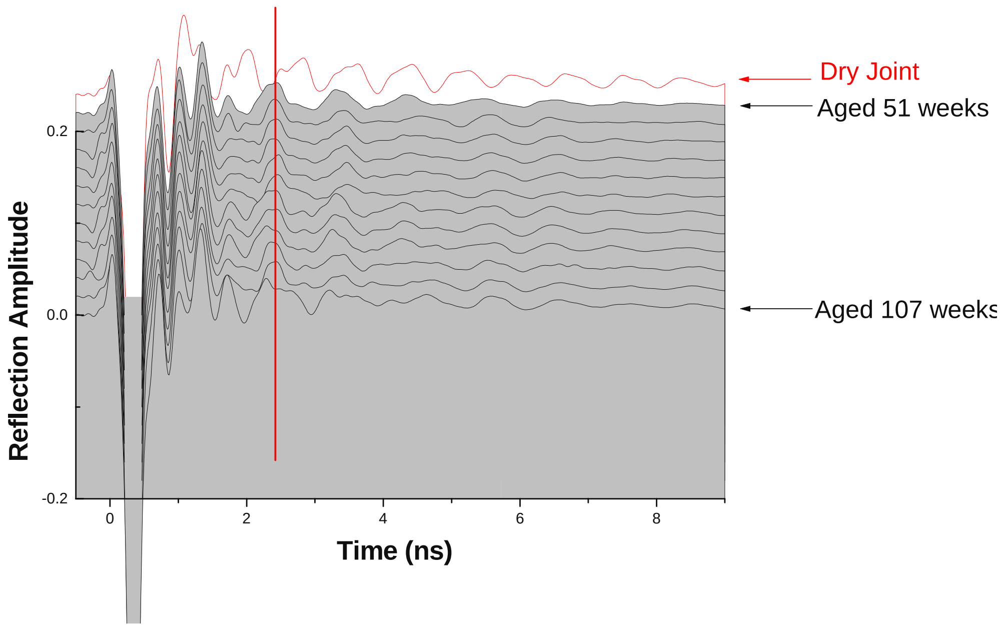

Figure 3 Time domain data for a 75°C aged Epibond joint, over the previous 12 months of its ageing cycle

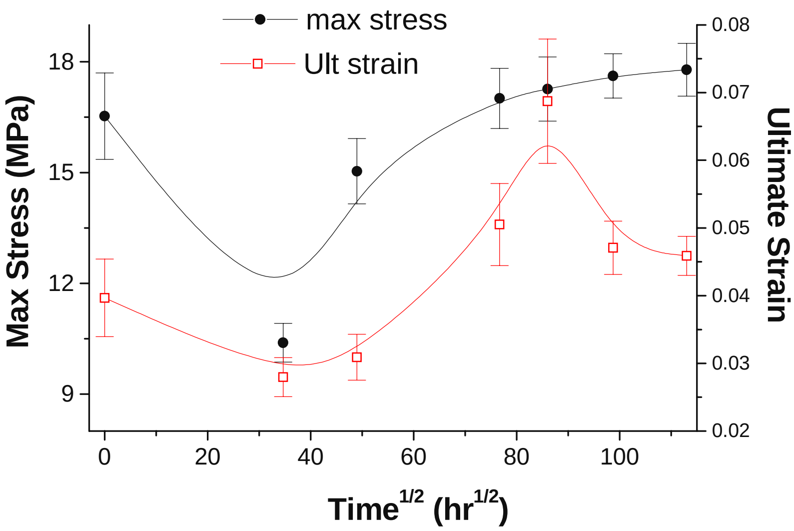

Figure 4 Average maximum stress and ultimate strain values for 50°C aged Epibond 1590 / aluminium joints.

Carbon Fibre composite/3M AF163

Carbon fibre composite plates bonded with 3M AF163 were immersed in water at 75°C and thermally spiked to -20°C every 14 days.

Because of the lack of corrosion it is possible to follow the absorption gravimetrically in this case and the results are shown in Figure 5 for both the frozen and unfrozen samples. Gravimetrically both sets appear to have reached an equilibrium water uptake but the dielectric data shows a different pattern of behaviour. The frequency

domain data shows a distinction between frozen and un-frozen samples at 10kHz (Figure 6) and 3 MHz (Figure 7), the frozen seeming to have reached an equilibrium value at long times whilst the un-frozen have not. Similar behaviour is seen in the time domain results. At long exposure periods the frozen joints (Figure 8) show an equilibrium in the first pulse transit time whereas the un-frozen joints (Figure 9) display an increasing transit time out to the longest exposure period, consistent with the frequency domain behaviour.
The dielectric response is showing a distinction between the two sets that the gravimetric measurements does not because the dielectric response is primarily measuring the change in the adhesive alone. The gravimetric response is measuring the water uptake in CFRP plates and the adhesive, and presumably the CFRP plate uptake is the dominant feature.

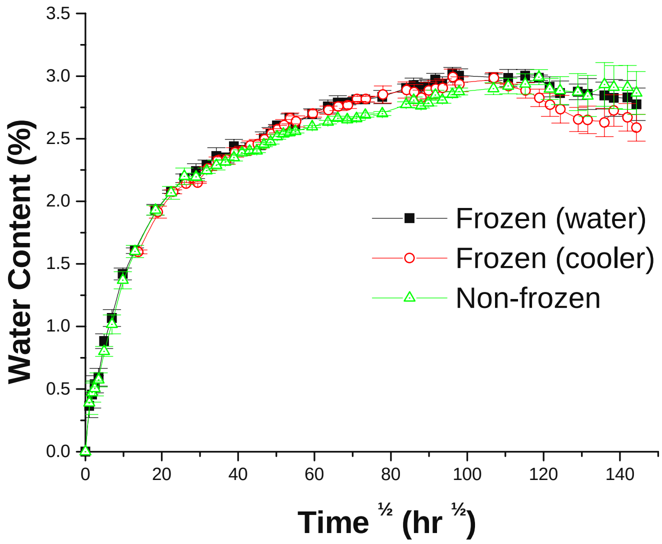

Figure 5 Changes in the weight of CFRP/AF 163-2K joints

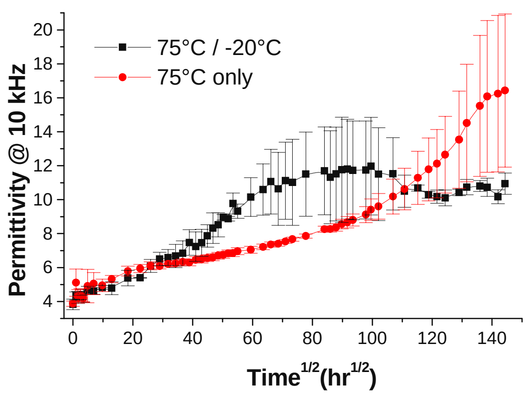

Figure 6 Comparison of dielectric ageing profiles of frozen and non-frozen CFRP joints at 10kHz

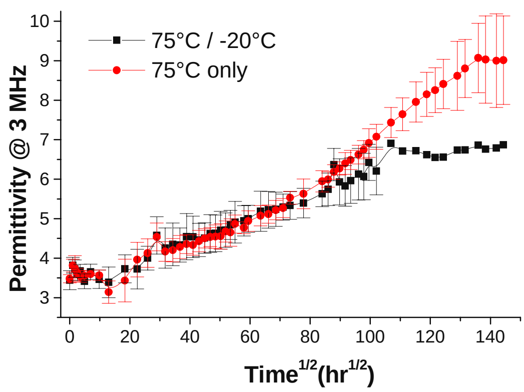

Figure 7 Comparison of dielectric ageing profiles of frozen and non-frozen CFRP joints at 3MHz

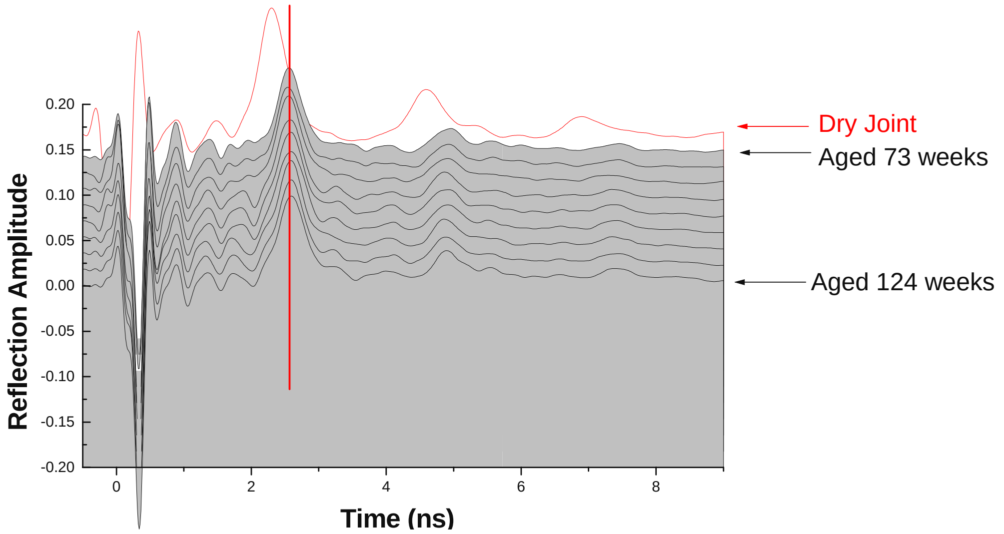

Figure 8 Time domain data for a frozen CFRP joint, over the previous 12 months of its ageing cycle

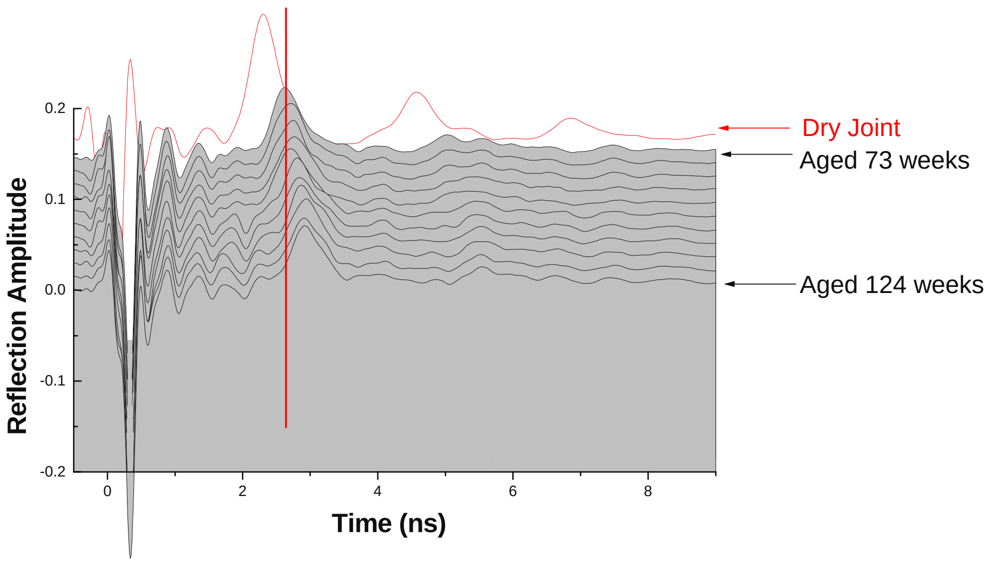

Figure 9 Time domain data for a non-frozen CFRP joint, over the previous 12 months of its ageing cycle.

Aluminium/3M AF163

Aluminium plates bonded with 3M AF163 were immersed in water at 75^{o}C with thermal spiking to -20^{o}C every 14 days.

In comparison to the Epibond 1590 joints the frequency domain data shows reasonably stable behaviour at long times, (Figure 10 and Figure 11). Consistent with this frequency domain behaviour, and again in contrast with the Epibond 1590 result the time domain behaviour (Figure 12) shows stability at long time exposure and no significant disruption of the pulse pattern at long exposure times.

The mechanical data also shows the stable long term behaviour after the expected decrease in strength with initial exposure (Figure 13). The correlation of maximum stress with permittivity value is shown in Figure 14.

The Aluminium/AF163 joints have undergone desorption and in this case it is possible to measure the weight loss, assumed indicative of the water loss from the adhesive, whereas in the absorption experiment the corrosion of the aluminium produces weight changes which far exceed the weight increase due to water uptake in the adhesive. The weight loss is shown in Figure 15 and the corresponding fall in permittivity shown in Figure 16 and Figure 17.There is now a very good correlation between the fall in permittivity and the weight loss (Figure 18 and Figure 19) and a consistency between the greater permittivity change and weight loss for the joints that underwent thermal spiking to -20C.
The time domain data of the desorption (Figure 20 and Figure 21) show a recovery of the shorter transit time as the water is lost and a good recovery of the pulse pattern. It remains to be seen if the mechanical test data shows a corresponding improvement in the mechanical properties

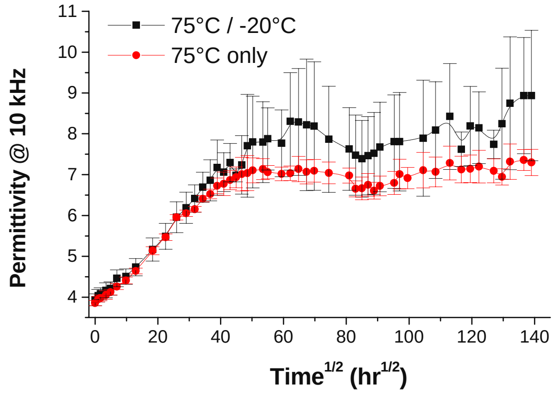

Figure 10 Comparison of dielectric ageing profiles of frozen and non-frozen joints at 10 kHz

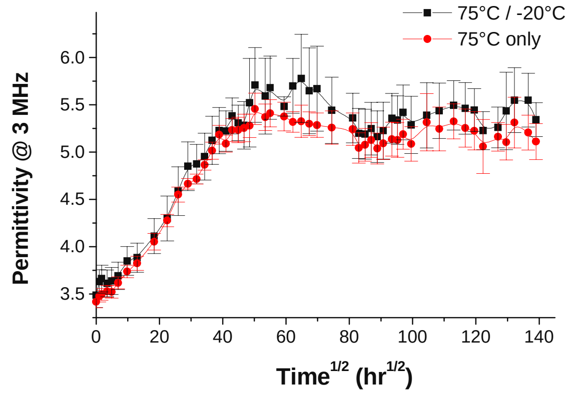

Figure 11 Comparison of dielectric ageing profiles of frozen and non-frozen joints at 3 MHz.

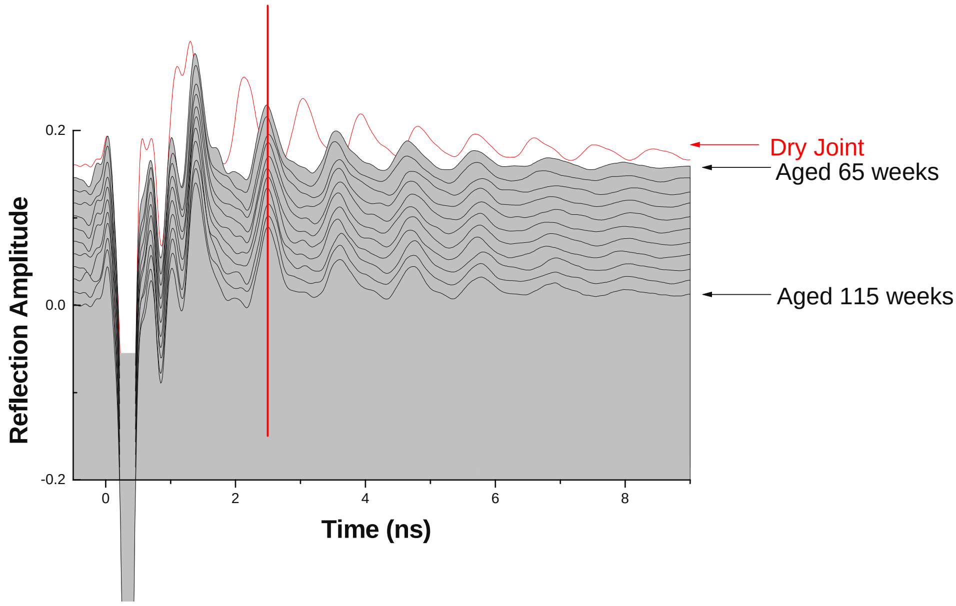

Figure 12 Time domain data for a non-frozen aluminium joint, over the final 12 months of its ageing cycle.

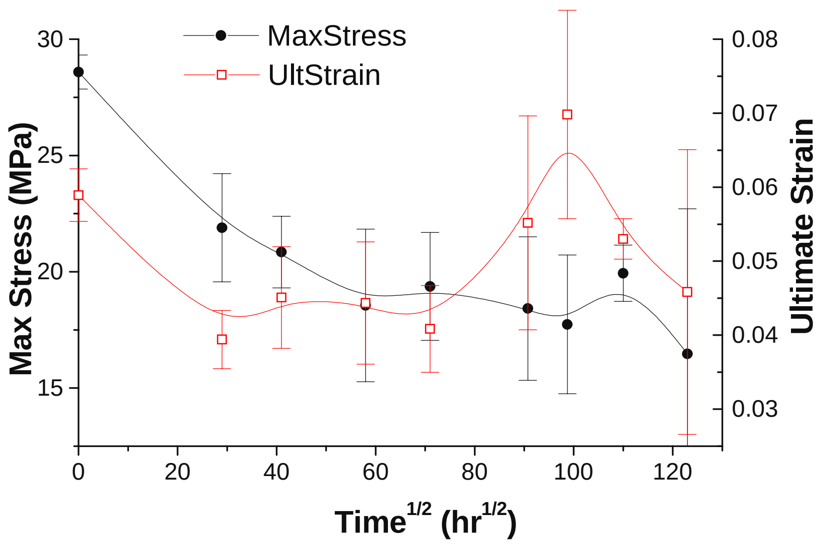

Figure 13 Average maximum stress and ultimate strain values for frozen aluminium joints.

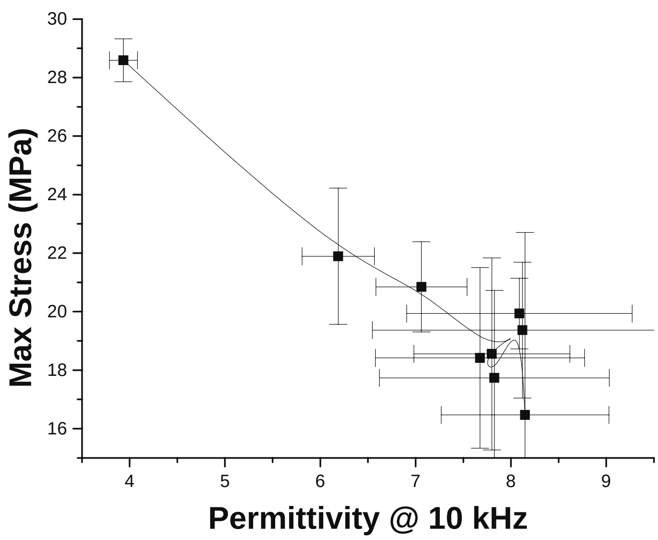

Figure 14 Correlation of maximum stress and permittivity for aluminium/AF 163-2K joints

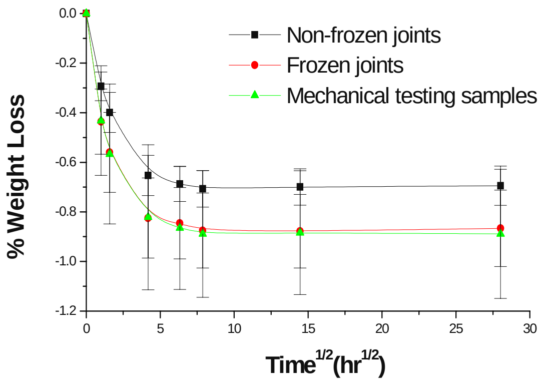

Figure 15 Change in weight of desorbed aluminium/AF 163-2K joints.

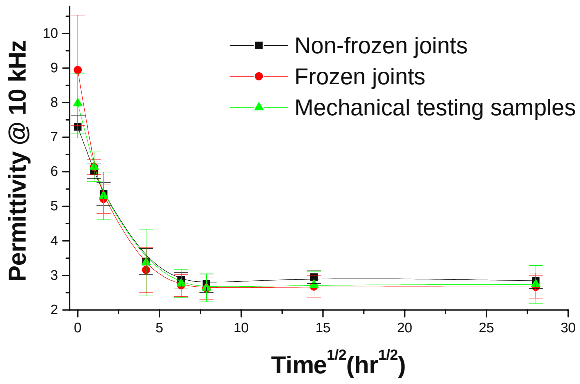

Figure 16 Permittivity at 10 kHz of desorbed joints

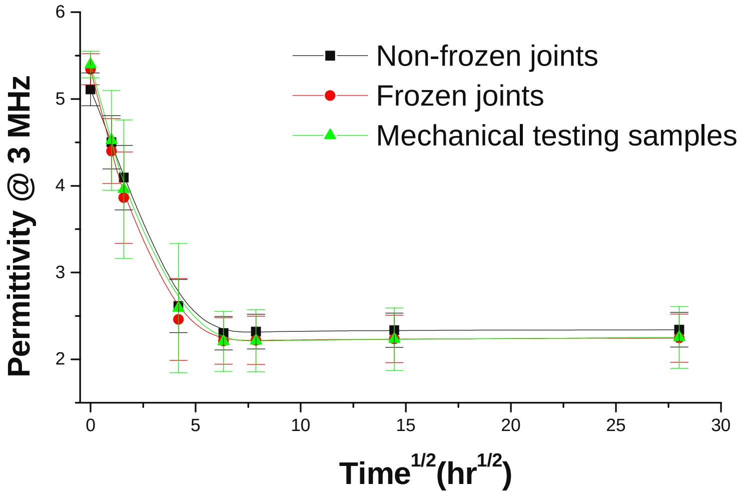

Figure 17 Permittivity at 3 MHz of desorbed joints

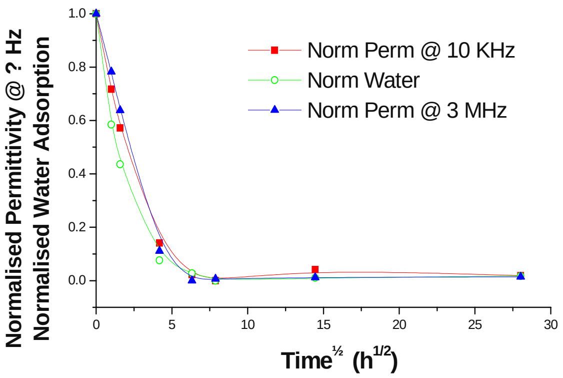

Figure 18 normalised water content and permittivity for frozen joints

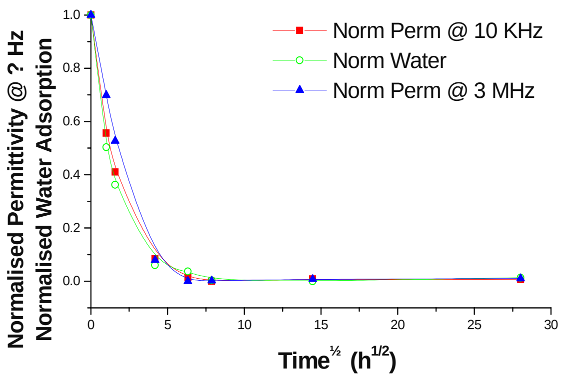

Figure 19 normalised water content and permittivity for un-frozen joints

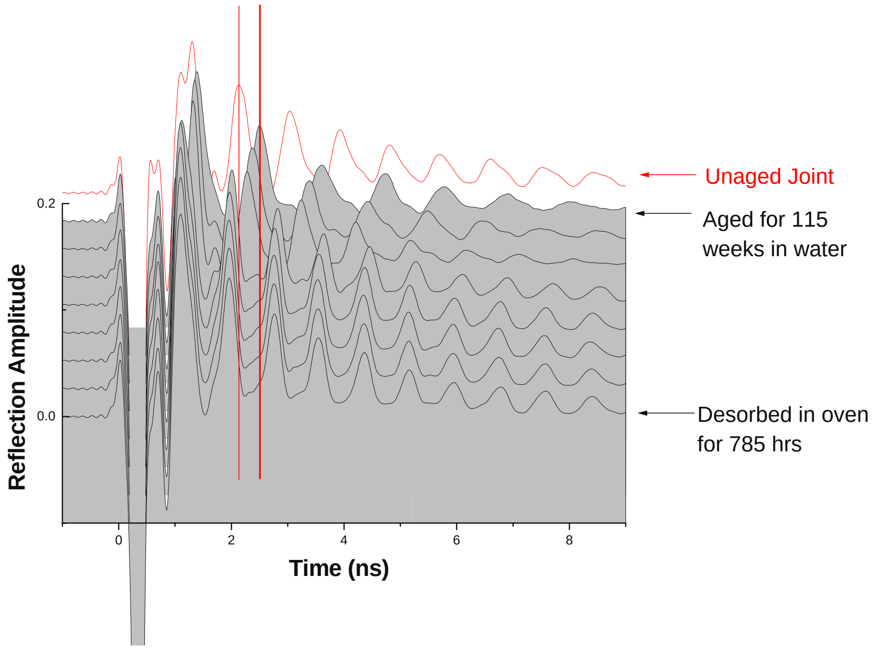

Figure 20 Time domain response for desorption of non-frozen aluminium joints.

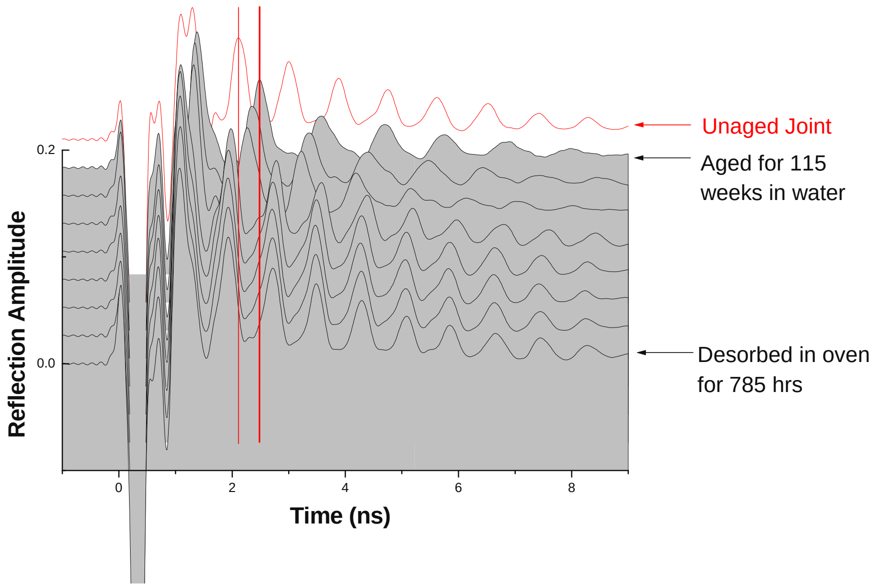

Figure 21 Time domain response for desorption of frozen aluminium joints.

2. Adhesive

The disks of Vantico Epibond 1590 which were earlier exposed to water at 50^{o}C and 75^{o}C have now undergone desorption. (Figure 22) and (Figure 23). Interestingly the 75^{o}C exposed material shows a net weight loss implying that some material has leached out during the exposure, again consistent with problems in the cure cycle of this material. There is an agreement between the fall in weight and the fall in permittivity as the systems undergo desorption (Figure 24) and (Figure 25).
The Dynamic Mechanical Analysis of the desorbed samples has not yet been completed.

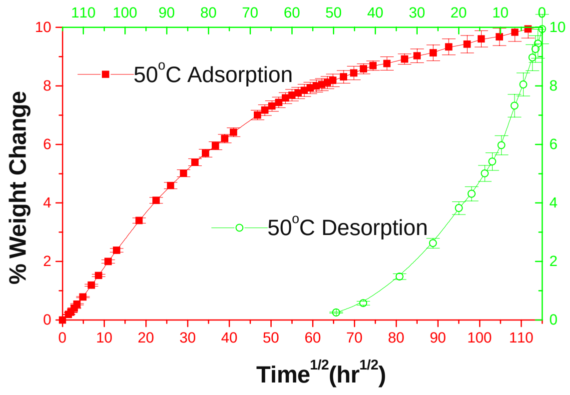

Figure 22 Changes in the weight over the adsorption/desorption cycles for 50°C and (b) & aged Epibond 1590 adhesive

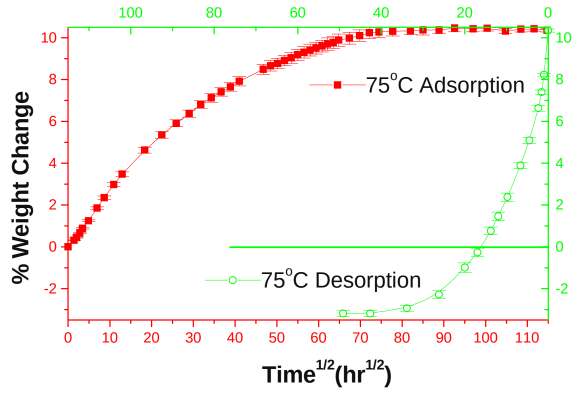

Figure 23 Changes in the weight over the adsorption/desorption cycles for 75°C aged Epibond 1590 adhesive

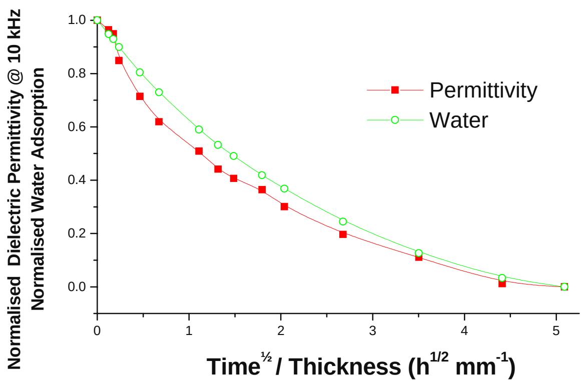

Figure 24 Normalised permittivity and water content data at 10 kHz for 50°C desorbing Epibond 1590 adhesive.

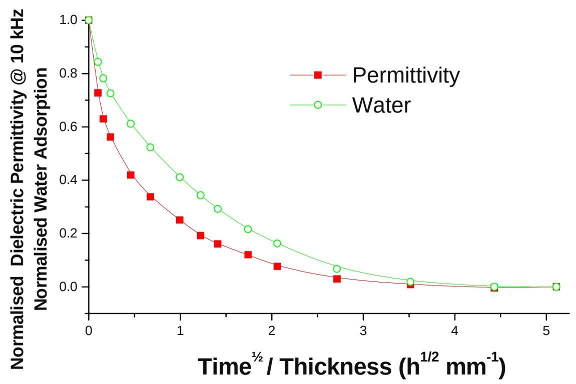

Figure 25 Normalised permittivity and water content data at 10 kHz for 75°C desorbing Epibond 1590 adhesive.

3a. Aluminium Oxides

The FTIR DRIFTS (**D**iffuse **R**eflectance **I**nfra **R**ed **F**ourier **T**ransmission) has not proved a practical way of measuring water uptake in aluminium oxide powders and so an alternative method based on glancing angle FTIR method is being use. It has been shown in the literature (*Ageing of aluminium oxide surfaces and their subsequent reactivity towards bonding with organic functional groups*, van den Brand J, Van Gils S, Beentjes PCJ, Terryn H, de Wit JHW, APPLIED SURFACE SCIENCE, **235** 465-474, 2004)
that the latter method will allow direct measurement of changes in hydration of the oxide layer on aluminium plates.

These FTIR measurements will be made in conjunction with an alternative high frequency dielectric measurement which should allow direct measurement of the free oxide layer on anodised aluminium plates.

4 Mathematical modelling

The objective of this work is two fold, to demonstrate

- the information from dielectric measurements of adhesive joints, particularly at high frequencies, correlates with the mechanical strength of the joints.
- it is possible to make dielectric measurements of sufficient accuracy on mechanical test coupons and ultimately in service structures.

The computational part of this work is concerned with the second objective which amounts to the development of a robust method of correcting for the effects of joint thickness variation and variations in the connector coupling behaviour. The co-operation with Prof Sean McKee's group in the Mathematics department has, as reported earlier, yielded a more robust iterative method of solving for the permittivity than the earliest method employed but one limitation has become apparent.

The problem is essentially solving for permittivity in the following given the experimentally measured reflection coefficient.

reflection coefficient = FN[connector (4 parameters),
joint geometry (?parameters),
permittivity (?parameters),
adherend resistivity (1 parameter)]

The method developed iterates to a solution for (constrained) variations in all the parameters which is great improvement in the previous method which required fixed values everything except the permittivity. The sensitivity of the solution to the conductor resistivity is however poor compared to the other parameters and so unrealistic values of adherend conductivity result.
Since there is evidence that the apparent adherend conductivity is a function of the state of the aherend/adhesive interface it is important that we are able to recover this quantity. Consequently we have developed an alternative iteration which is based on a partial separation of variables in the above equation. In this case by iterating on some of the parameters on the first pass and the remainder on a second pass we have been able to demonstrate that the adherend conductivity is recoverable.
Work is in progress to combine this latest approach with the method developed in conjunction with Prof McKee's group.

Papers arising

Dielectric and mechanical studies of the durability of adhesively bonded aluminium structures subjected to temperature cycling. Part 1: examination of moisture absorption
Proc. Inst. Mech. Eng. Pt. L-J. Mater.-Design Appl **218**, 169-182 2004
Pethrick RA, Armstrong GS, Banks WM, Crane RL, Hayward, D

Dielectric and mechanical studies of the durability of adhesively bonded aluminium structures subjected to temperature cycling. Part 2: examination of the failure process and effects of drying
Proc. Inst. Mech. Eng. Pt. L-J. Mater.-Design Appl **218**, 183-192 2004
Pethrick RA, Armstrong GS, Banks WM, Crane RL, Hayward, D